

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> : <b>A61K 7/027, 7/025, 7/48</b>	A1	(11) International Publication Number: <b>WO 96/40044</b> (43) International Publication Date: 19 December 1996 (19.12.96)
(21) International Application Number: PCT/US96/07122		(81) Designated States: CA, CN, JP, MX, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).
(22) International Filing Date: 17 May 1996 (17.05.96)		
(30) Priority Data: 08/481,776 7 June 1995 (07.06.95) US 08/545,976 20 October 1995 (20.10.95) US		<b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(71) Applicant: THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US).		
(72) Inventors: RABE, Thomas, Elliot; 6-N Tyler Falls Court, Baltimore, MD 21209 (US). DRECHSLER, Lee, Ellen; 3145 Bishop Street, Cincinnati, OH 45220 (US). SMITH, Edward, Dewey, III; 11092 Allenhurst Boulevard, West, Cincinnati, OH 45241 (US).		
(74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217 (US).		
(54) Title: TRANSFER-RESISTANT LIP COMPOSITIONS		
(57) Abstract		
<p>The present invention relates to stable lip compositions comprising a first material and a second material wherein the second material is entrapped throughout the first material. The first and second materials are sufficiently incompatible with each other wherein when the composition is applied to the lips, the second material separates from said first material forming a barrier layer over the first material deposited on the lips. Using such a composition eliminates having to individually apply a barrier layer over the lipstick previously applied to the lips.</p>		

***FOR THE PURPOSES OF INFORMATION ONLY***

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MW	Malawi
AT	Austria	GE	Georgia	MX	Mexico
AU	Australia	GN	Guinea	NE	Niger
BB	Barbados	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IE	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KE	Kenya	RO	Romania
BY	Belarus	KG	Kyrgyzstan	RU	Russian Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	KZ	Kazakhstan	SG	Singapore
CH	Switzerland	LJ	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SZ	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Latvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

## TRANSFER-RESISTANT LIP COMPOSITIONS

5

### TECHNICAL FIELD

The present invention relates to stable lip compositions comprising a first material and a second material wherein the second material is entrapped throughout the first material. The first and second materials are sufficiently incompatible with each other wherein when the composition is applied to the lips, the shearing forces created causes the second material to separate from the first material forming a barrier layer over the first material deposited on the lips. Using such a composition eliminates having to individually apply a barrier layer over the lipstick previously applied to the lips.

### BACKGROUND OF THE INVENTION

Lip treatment products whose primary purpose is to extend wear, improve the blot transfer resistance and heighten the gloss of said lipstick are well known in the art. Some of these products such as overcoats utilize a variety of polymeric fluids and film forming technologies to form a barrier layer that avoids transference of the lipstick it is applied over. Examples of such compositions are disclosed in Japanese Patent Application Number HEI 5[1993]-221829, published August 31, 1993 and copending U. S. Serial Number 08/361,246, filed December 21, 1994; both incorporated herein by reference. Although said overcoat products can be effective in preventing inadvertent transfer onto objects, they do require separate application over the pigmented lip composition applied to the lips. It is, therefore, advantageous to provide a product which combines these two separate application steps into one single step.

### SUMMARY OF THE INVENTION

The present invention is a lip composition comprising a first and second material, wherein the first material has a second material entrapped throughout it. Said first and second materials are sufficiently incompatible where upon application of the present composition to the lips, the shearing forces created causes the second material to separate from the first material forming a barrier layer over the first material deposited on the lips.

Therefore, an objective of the present invention is to provide a composition wherein the second material forms a barrier, thereby preventing inadvertent transference of the first material to objects such as tableware and clothing contacting the lips. Another object of the present invention is to provide a barrier layer over a pigmented lip composition in a single step. Still another object of the present invention is to provide a composition having desirable application and feel characteristics as well as improve luster and shine of the composition once applied to the lips. A last object of the present invention is to provide processing steps for making such compositions.

The composition of the present invention can take a number of forms often associated with lip composition, including solid sticks, creams and balms. Unless otherwise indicated, all percentages disclosed herein are by total weight of the composition.

#### DETAILED DESCRIPTION OF THE INVENTION

5        1. First Material

The first material or continuous material of the present composition comprises lipophilic materials selected from the group consisting of waxes, volatile oils, nonvolatile oils and mixtures thereof wherein the oils are compatible with said wax when said wax is liquified. The first material comprises from about 30% to about 95%, preferably from about 50% to about 85%, and most 10        preferably from about 50% to about 60% of the composition.

Waxes act as solidifying agents thereby assisting in forming solid structures such as "bullet" shaped lipsticks. Waxes as used herein are defined as organic compounds or mixtures of high molecular weight substances, that are thermoplastic, forming a solid mass at ambient temperature/room temperature. As used herein wax refers to single type of wax or mixtures of 15        waxes.

Said waxes include hydrocarbons or esters of fatty acids and fatty alcohols and are derived from natural, synthetic and mineral sources. Such waxes are disclosed in Warth, Chemistry and Technology of Waxes, Parts 1 and 2, 1956, Reinhold Publishing Corporation. Natural waxes can be of animal origin, such as beeswax, spermaceti, lanolin, shellac wax, of vegetable origin, e.g. 20        carnauba, candelilla, bay berry, sugar cane wax, or of mineral origin, e.g. ozokerite, ceresin, montan, paraffin, microcrystalline wax, petroleum and petrolatum wax. Synthetic waxes include polyol ether-esters such as carbowax and hydrocarbon-type waxes, silicone waxes and polyethylene wax having melting points greater than about 40°C. The waxes most useful herein have melting points from about 55°C to about 110°C and are selected from the C<sub>8</sub> to C<sub>50</sub> 25        hydrocarbon waxes.

The waxes preferred for use in the present compositions are selected from the group consisting of candelilla, beeswax, beeswax having free fatty acids removed (modified beeswax), carnauba, spermaceti, montan, ozokerite, ceresin, paraffin, bayberry, castor waxes, synthetic waxes, microcrystalline waxes, silicone waxes (modified to be compatible with other first 30        materials) and mixtures thereof. More preferably the waxes are selected from the group consisting of microcrystalline, spermaceti, candelilla, modified beeswax, carnauba, ozokerite, paraffin, ceresin, silicone waxes and mixtures thereof. Most preferably, the waxes are selected from the group consisting of candelilla, ozokerite, paraffin, carnauba wax and mixtures thereof.

The volatile and nonvolatile oils of the present invention have a number of functional 35        purposes, for example application, adhesion, yield, gloss and perhaps most importantly occlusive moisturization generally. Said oils are liquid at ambient temperature and include esters, triglycerides, hydrocarbons, silicones and mixtures thereof.

Volatile oils, particularly those selected from the group consisting of volatile hydrocarbons, polydimethylsiloxanes, cyclic polydimethyl siloxane and mixtures thereof are incorporated into the lipstick compositions to provide more rigid lipstick films due to higher wax to oil ratio in the film once the volatile component has evaporated. Particularly useful are the 5 volatile oils that are hydrocarbon based. Said volatile oils are selected from the group consisting of saturated branched hydrocarbons, unsaturated branched hydrocarbons, saturated linear hydrocarbons, unsaturated linear hydrocarbons and mixtures thereof having from about from about 8 to about 30 carbon atoms per molecule, preferably from about 8 to about 20 carbon atoms per molecule, and most preferably from about 8 to about 14 carbon atoms per molecule. The 10 rigid film produced by the addition of a volatile oil reduces the amount of lipstick that is transferred to other objects. In spite of the teaching in the art, such as European Patent Publication 602905, published June 22, 1994, regarding the use of volatile oils to make rigid films, the present invention is practically better. The present invention delivers a barrier layer over said rigid film thereby providing the user with a long lasting moist and lubricious feeling on 15 the lips which the rigid film alone cannot create.

As previously mentioned, this invention also envisions the addition of nonvolatile oils. Specific nonvolatile oils useful in the present invention include caprylic triglycerides; capric triglycerides; isostearic triglycerides; adipic triglycerides; wheat germ oil; hydrogenated vegetable oils; petrolatum; branched-chain hydrocarbons; alcohols and esters; castor oil; lanolin oil; corn oil; cottonseed oil; olive oil; palm kernel oil; rapeseed oil; safflower oil; jojoba oil; evening primrose oil; avocado oil; mineral oil; sheabutter; octylpalmitate; maleated soybean oil; glycerol trioctanoate; diisopropyl dimerate; volatile and non-volatile silicone oils including dimethicone, phenyl dimethicone, cyclomethicone, poly(perfluoroalkyl) siloxanes, linear and cyclic polyalkyl siloxanes and mixtures thereof. Preferable oils used in the present invention are selected from the 20 group consisting of caprylic triglycerides, capric triglycerides, isostearic triglyceride, castor oil, adipic triglyceride, dimethicone, octyl dodecanol, oleyl alcohol, hydrogenated vegetable oils, maleated soybean oil, lanolin oil, polybutene, oleyl alcohol; hexadecyl alcohol; wheat germ glycerides and mixtures thereof. The individual oils or the aggregate of the oils selected should be soluble with the liquefied waxes selected for use herein. Therefore, oils have a solubility 25 parameter from about 5 to about 10. The respective solubility parameters for waxes and oils are reported in "Cosmetics & Toiletries", Vol 103, October 1988; incorporated herein by reference.

Emollients useful in the present invention are found in The C.T.F.A. Cosmetic Ingredient Handbook, pages 572-575, 1992; herein incorporated by reference. Said emollients include lanolin, synthetic lanolin derivatives, modified lanolins, isopropyl palmitate, isononyl 30 isononanoate, isopropyl isostearate, cetyl ricinoleate, octyl palmitate, cetyl ricinoleate, glyceryl trioctanoate, diisopropyl dimerate, propylene glycol, myristyl acetate, isopropyl myristate, diethyl sebacate; diisopropyl adipate; tocopheryl acetate; tocopherol linoleate; hexadecyl stearate; ethyl

lactate; cetyl lactate, cetyl oleate, octyl hydroxystearate; octyl dodecanol, decyl oleate, propylene glycol ricinoleate, isopropyl lanolate, pentaerythrityl tetraesteareate, neopentylglycol dicaprylate/dicaprate, hydrogenated coco-glycerides, isotriidecyl isononanoate, isonyl isononanoate, myristal myristate, triisocetyl citrate, cetyl alcohol, octyl dodecanol, oleyl alcohol and mixtures thereof. Particularly useful emollients are selected from the group consisting of lanolin, isopropyl isostearate, cetyl lactate, octyl hydroxystearate and mixtures thereof.

Humectants useful in the present invention include those as disclosed in The C.T.F.A. Cosmetic Ingredient Handbook, page 567, 1992; herein incorporated by reference. Occlusives useful in the present invention are likewise found in the C.T.F.A. Cosmetic Ingredient Handbook, at pages 578-580; herein incorporated by reference.

## 2. Second Material

The second material of the present invention is selected from the group consisting of wherein said second material is a volatile materials having a vapor pressure of less than about 10mm Hg at 30°C, non-volatile materials and mixtures thereof wherein said second material as a viscosity from 15 about 0.5 to about 5,000 centistokes. In the present invention, the second material comprises from about 5% to about 70%, preferably 15% to about 50%, and most preferably from about 30% to about 40% of the composition.

The second material of the present invention comprises a single material or an aggregate of materials which are incompatible with the first material. By incompatible it is meant that the second material is dispersed throughout the liquefied first material, similar to an emulsion-phase. Upon solidification, this second material eventually is entrapped within the first material, preferably in an even distribution; i.e. small particles or droplets distributed throughout the first material. The second material remains entrapped within the first material until the composition experiences shearing forces created by application of the composition on the lips. At this point the entrapped second material has 25 no physical barriers to prevent its coalescence wherein the second material spreads over the first material, essentially forming a barrier layer over the first material.

It should be noted, however, some of the individual components making up the first and second materials may exhibit some degree of compatibility with components of the opposite material. In fact, some of the materials selected as either the first or second material may partition between the 30 first and second materials. Volatile oils are one such example of a component that may partition between the first and second materials. However, in the present invention any component demonstrating at least some compatibility for both the first and second materials will not exhibit compatibility to the degree that its inclusion makes the first and second materials completely incompatible. Furthermore, while the second material is incompatible with the first material, said 35 second material may be an aggregate of materials wherein some and possibly all of those materials are incompatible with each other.

The existence of a second material entrapped within said first material is readily detectable

by an artisan using routine analytical methods including centrifugation of the emulsion wherein two separate layers form, microscopy of solid compositions wherein the entrapped second material is directly observed within the solid lipophilic material continuous phase.

By "volatile" it is meant that the vapor pressure of said volatile oil is less than about 10mm Hg, preferably less than about 1mm Hg, and most preferably less than about 0.5mm Hg at 30°C as measured using analytical means known to those skilled in the art. Said second material of the present invention has a viscosity from about 0.5 to about 5,000 centistokes (cSt), preferably from about 1 to about 500 cSt, most preferably from about 10 to about 100 cSt. Although the second material of the present invention may be either volatile or non-volatile, non-volatile materials are preferred.

Said second material of the present invention preferably is close to being optically transparent or translucent at the thickness that results from application of the said composition to the lips. The second material which forms the barrier layer or film over the first material on the lips is thus undetectable to the naked eye after the composition is applied to the lips so as not to hinder the presentation of the color of the present composition on the lips. This optical transparency is measured in terms of a film contrast ratio. The contrast ratio is preferably between 0.00 and 0.20 for a 1 mil (0.001 inch) thick film of the said second material. The contrast ratio measurement technique is as follows:

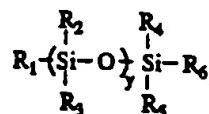
1. Place a Type 2A Opacity Chart (The Leneta Company, Ho-Ho-Kus, NJ, USA) onto a flat vacuum table;
- 20 2. Draw down a 0.001" thick film of the said second material using a 0.001" Byrd Film Applicator (MCD Industries, Medfield, MA, USA) onto the opacity chart, coating both the white and black regions of the chart; and
3. Measure the Y-value over the black region and the Y-value over the white region of the chart using a spectrophotometer (e.g. Microflash, Datacolor International, Lawrenceville, NJ, USA).

The contrast ratio is defined as Y-value (black) / Y-value (white).

As previously mentioned the second material forms a barrier layer over the first material after the composition is applied to the lips. Therefore, it is critical that the second material separates from the first material and easily spreads over the lips by routine application of the composition to form said barrier layer without receding back into the first material or forming individual droplets over the first material. The ability of a second material to spread and remain in such a state is determined by the surface tension of the second material and by the surface tension of the underlying first material. The second material is found to be spreadable over a surface when its surface tension is equal to or below the critical surface tension for wetting of the surface. The critical surface tension for wetting a surface, first defined by Fox and Zisman, is equal to the surface tension of the second material which just exhibits a zero contact angle on the surface; see H.W. Fox and W.A. Zisman, J. Colloid Sci., 5, 514 (1950), H.W. Fox and W.A. Zisman, J. Colloid Sci., 7, 109 (1952) and H.W. Fox and W.A.

Zisman, J. Colloid Sci., 7, 428 (1952), all incorporated herein by reference. Since this critical surface tension of wetting can vary with composition of the first material, a second material with a surface tension equal to or below the critical surface tension of wetting must be chosen for each particular first material composition. Such a second material would exhibit a contact angle of about zero when placed 5 on a flat, drawn-down film of the composition's first material. The surface tension of the second material of the present invention is less than about 35 dynes/cm, preferably less than about 30 dynes, most preferably less than about 25 dynes/cm.

Second materials useful in the present invention include poly(organosiloxane) fluids conforming to the formula:



10

wherein the end groups  $R_1$  and  $R_6$  are independently selected from the group consisting of hydroxyl groups, lower alkyl groups having carbon chain lengths from about  $C_1$  to about  $C_6$  and mixtures thereof, preferably methyl groups and the non-end groups  $R_2$ ,  $R_3$ ,  $R_4$  and  $R_5$  are independently selected from methyl groups, fluoroalkyl groups, phenyl groups and mixtures thereof.

15

The poly(organosiloxane) fluids with non-end groups ( $R_2$ ,  $R_3$ ,  $R_4$  and  $R_5$ ) comprising methyl groups are known in the art and provide the final product with a relatively non-lipophilic character. Commercially available non-volatile silicone fluids having such non-end groups include those available from Dow Corning as the 200 Fluids, and those available from General Electric as SF-96 Series.

20

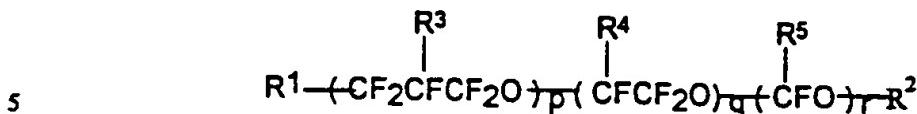
Silicone fluids with non-end groups comprising fluoroalkyl groups are also useful herein. It is preferable, however, that the fluorine atom is attached to alkyl groups having a  $C_3$  to  $C_8$  chain length wherein the fluorine atom is attached to said alkyl group at a point no closer than third carbon atoms from the silicone/carbon bond. Commercially available non-volatile silicone fluids having such non-end groups include those available from Dow Corning as the 1265 Fluid series, and 25 those available from General Electric as the SF- 1153 Series, most preferred is the 1265 Fluid Series, most preferably those of having a viscosity from about 100cSt to about 350cSt.

Silicone fluids with the non-end groups comprising allyl groups are also useful in the present invention. The allyl groups which are particularly useful in the present invention are phenyl groups. Particularly useful allyl-substituted silicone fluids commercially available are available as the 556 30 Series from Dow Corning.

Preferable poly(organosiloxane) fluids of the present invention are selected from the group consisting of poly(dimethylsiloxane) fluids, poly(phenylmethylsiloxane) fluids, poly(fluoroalkylmethylsiloxane) fluids, the copolymers of said fluids and mixtures thereof. More preferred fluids are selected from the group consisting of poly(dimethylsiloxane) fluids, their 35 copolymers and mixtures thereof. Most preferred are poly(dimethylsiloxane) fluids and their

copolymers, preferably selected from the group consisting of dimethicone, phenyl dimethicone, phenyl trimethicone and mixtures thereof.

Non-silicone fluids also useful as a second material of the present invention include perfluoropolyethers of general formula:



wherein R<sup>1</sup> though R<sup>5</sup> are selected from the group consisting of fluorine atoms, perfluoroalkyl groups, oxyperfluoroalkyl groups and mixtures thereof, the value of p, q, and r collectively are of a value such that the perfluoropolyether molecular weight is from about 500 to about 10,000 wherein p, q and r may be equal. A preferred perfluoropolyether is the commercially available product known as Fomblin HC-

10 01,

-02, -03 and -04, HC-25 and HC-R available from Montefluosu of Milano, Italy. Non-volatile perfluoropolyethers are preferred.

### C. Optional Ingredients

Compositions of the present invention further include optional ingredients which may be added to the composition disclosed above to provide various consumer desirable characteristics to the product. Said optional ingredients include those routinely used in the cosmetic arts to produce a specific cosmetic effect which is deemed desirable.

Surfactants may be used in the present invention insofar as they do not sufficiently stabilize the second material entrapped within the first material or allowing said second material to separate from the first material upon application to the lips. Surfactants are well known to those skilled in the art of lipstick making in order to enhance dispersability of pigments and other solid materials like mica and talc, stabilize liquid dispersed phases such as water, glycerine and glycols, provide skin benefits such as emolliency, and skin feel modifiers; i.e. the right combination can make the stick less hard. Anionic surfactants, nonionic surfactants, cationic surfactants, amphoteric surfactants and mixtures thereof are suitable for use. The more common surfactants used in the present invention include those found in the C.T.F.A. Cosmetic Ingredient Handbook, pages 587-592, 1992; herein incorporated by reference. Some of the more commonly used ones include ethoxylated castor oil, lecithin, fatty acids and salts of fatty acids (sodium stearate, stearic acid, oleic acid, potassium stearate, zinc stearate), fatty alcohols (oleyl alcohol, e.g.), ascorbyl palmitate, oxidized waxes, mono and di-glycerides (glyceryl oleate), lauroyl lysine, cetyl lactate and mixtures thereof.

In addition to surfactants other ingredients such as preservatives, sunscreens, UV absorbers, anti-oxidants, flavorings, perfumes, colorants, dyes and other ingredients routinely used in the art. In the case of dyes and colorants which are incorporated into the first material, it is important that the first and second materials are sufficiently incompatible to avoid tinting or coloring the second material.

## EXAMPLES

**Example 1:**

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Candelilla Wax	4.70
Carnauba Wax	3.40
Ozokerite	4.30
Paraffin	2.10
Lanolin	4.30
Isopropyl Isostearate	12.80
Cetyl Lactate	1.70
Octyl Hydroxystearate	8.50
Ascorbyl Palmitate	0.70
Propyl Paraben	0.16
Vitamin E Acetate	0.04
Castor Oil	34.00
Red #7 Ba Lake	2.60
Red #6 Ca Lake	2.10
Hematite	4.70
Titanium Dioxide	8.90
Perfluoropolyether*	5.00

\* available as Fomblin HC/25 from Ausimont SPa

5   **Example 2:**

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Candelilla Wax	4.20
Carnauba Wax	3.10
Ozokerite	3.80
Paraffin	1.90
Lanolin	3.80
Isopropyl Isostearate	11.50
Cetyl Lactate	1.50
Octyl Hydroxystearate	7.60
Ascorbyl Palmitate	0.70
Propyl Paraben	0.06
Vitamin E Acetate	0.04
Castor Oil	30.40
Red #7 Ba Lake	2.30

Red #6 Ca Lake	1.90
Hematite	4.20
Titanium Dioxide	8.00
10 cSt Dimethicone*	15.00

\* available as Dow Corning 200 Fluid

**Example 3:**

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Candelilla Wax	3.50
Carnauba Wax	2.50
Ozokerite	3.20
Paraffin	1.60
Lanolin	3.20
Isopropyl Isostearate	9.30
Cetyl Lactate	1.30
Octyl Hydroxystearate	6.30
Ascorbyl Palmitate	0.50
Propyl Paraben	0.07
Vitamin E Acetate	0.03
Castor Oil	25.00
Red #7 Ba Lake	1.90
Red #6 Ca Lake	1.50
Hematite	3.50
Titanium Dioxide	6.6
50 cSt Dimethicone*	30.00

\* available as Dow Corning 200 Fluid

**Example 4:**

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Candelilla Wax	4.00
Beeswax	2.00
Ozokerite	3.20
Paraffin	1.60
Lanolin	3.20
Isopropyl Isostearate	9.30
Cetyl Lactate	1.30
Octyl Hydroxystearate	6.30

Ascorbyl Palmitate	0.50
Propyl Paraben	0.07
Vitamin E Acetate	0.03
Castor Oil	25.00
Red #7 Ba Lake	1.90
Red #6 Ca Lake	1.50
Hematite	3.50
Titanium Dioxide	6.60
50 cSt Dimethicone*	15.00
Perfluoropolyether**	15.00

\* available as Dow Corning 200 Fluid  
 \*\* available as Fomblin HC/25 from Ausimont SPa

**Example 5:**

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Candelilla Wax	3.00
Carnauba Wax	3.00
Ozokerite	4.80
Lanolin	3.20
Isopropyl Isostearate	9.30
Cetyl Lactate	1.30
Octyl Hydroxystearate	6.30
Ascorbyl Palmitate	0.50
Propyl Paraben	0.07
Vitamin E Acetate	0.03
Castor Oil	25.0
Red #7 Ba Lake	1.90
Red #6 Ca Lake	1.50
Hematite	3.50
Titanium Dioxide	6.60
50cSt Phenylmethicone*	30.00

\* available as Dow Corning 556 Fluid

5

**Example 6:**

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Candelilla Wax	2.70
Carnauba Wax	2.00

Ozokerite	2.50
Paraffin	1.30
Lanolin	2.50
Isopropyl Isostearate	7.40
Cetyl Lactate	1.00
Octyl Hydroxystearate	4.90
Ascorbyl Palmitate	0.40
Propyl Paraben	0.05
Vitamin E Acetate	0.05
Castor Oil	19.70
Red #7 Ba Lake	1.50
Red #6 Ca Lake	1.20
Hematite	2.70
Titanium Dioxide	5.10
50cSt Dimethicone*	45.00

\* available as Dow Corning 200 Fluid

**Example 7:**

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Candelilla Wax	2.70
Carnauba Wax	2.00
Ozokerite	2.50
Paraffin	1.20
Lanolin	2.50
Isopropyl Isostearate	7.40
Cetyl Lactate	1.00
Octyl Hydroxystearate	4.90
Ascorbyl Palmitate	0.40
Propyl Paraben	0.15
Vitamin E Acetate	0.05
Castor Oil	25.00
Red #7 Ba Lake	1.50
Red #6 Ca Lake	1.20
Hematite	2.70
Titanium Dioxide	4.80
50 cSt Dimethicone*	20.00
50 cSt Phenylmethicone**	20.00

\*available as Dow Corning 200 Fluid

\*\*available as Dow Corning 556 Fluid

**Example 8:**

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Candelilla Wax	2.70
Carnauba Wax	2.00
Ozokerite	2.50
Paraffin	1.20
Lanolin	2.50
Isopropyl Isostearate	7.50
Cetyl Lactate	1.00
Octyl Hydroxystearate	4.90
Ascorbyl Palmitate	0.40
Propyl Paraben	0.05
Vitamin E Acetate	0.05
Castor Oil	19.70
Red #7 Ba Lake	1.50
Red #6 Ca Lake	1.20
Hematite	2.70
Titanium Dioxide	5.10
300cSt Fluorosilicone*	45.00

\*available as Dow Corning 1265 Fluid

**5 Example 9:**

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Candelilla Wax	1.50
Carnauba Wax	1.60
Ozokerite	1.30
Paraffin	0.70
Lanolin	1.40
Isopropyl Isostearate	4.20
Cetyl Lactate	0.50
Octyl Hydroxystearate	2.00
Ascorbyl Palmitate	0.20
Propyl Paraben	0.04
Vitamin E Acetate	0.06

Castor Oil	10.70
Red #7 Ba Lake	0.80
Red #6 Ca Lake	0.70
Hematite	1.50
Titanium Dioxide	2.80
50cSt Dimethicone*	70.00

( 99.5% total)

\* available as Dow Corning 200 Fluid

Examples 1-9 are assembled according to the following instructions:

Combine the pigments and castor oil in a vessel with stirring until incorporating the pigments into said castor oil. Pass the mixture through a 3-roll mill three times yielding a dispersion of pigment particles in oil; hereinafter referred to as the slurry.

- 5        Except for the second material, combine all the remaining ingredients in a vessel equipped with heating and mixing. Heat the combination to about 95°C, holding there for a sufficient time wherein said ingredients are in a liquid form. While maintaining the heat, stir the combination of ingredients for about 30 minutes, obtaining a transparent yellow fluid. Cool the combination to about 21°C until forming a pale yellow waxy solid; hereinafter referred to as the base.
- 10      Combine the pigment slurry and the base, hereinafter collectively referred to as the first material, with the second material in order of the highest weight percentage of the composition first. Heat this combination to about 90°C until the combination becomes liquid. Cease heating the combination and mix for about 3 minutes at 10,000 RPM using a Janke & Kunkel Ultra-Turrax turbine mixer. Pour the combination immediately into a room temperature aluminum bullet mold. Cool the filled mold until the combination is solid. Remove the solid composition from the molds and place in suitable cosmetic packaging.
- 15

Example 10:

<u>Ingredient</u>	<u>Weight Percent (%)</u>
Ceresin Wax	2.70
Carnauba Wax	2.00
Microcrystalline Wax	2.50
Paraffin	1.20
Tricostanyl PVP	2.50
Isoeicosane	7.50
Glyceryl Linoleate	1.00
Octyl Methoxycinnamate	4.90
Ascorbyl Palmitate	0.40
Propyl Paraben	0.05

Vitamin E Acetate	0.05
Red #7 Ba Lake	1.50
Red #6 Ca Lake	1.20
Hematite	2.70
Titanium Dioxide	5.10
50 CSt Dimethicone*	19.70
Perfluoropolyether**	45.00

\* available as Dow Corning 200 Fluid

\*\*available as Fomblin HC/4 from Ausimont SPa

Example 10 is made according to the following instructions:

Combine the pigments and dimethicone in a vessel equipped with stirring. After incorporating the pigments in the dimethicone, pass the mixture through a 3-roll mill three times  
5 yielding a dispersion of pigment particles in dimethicone; hereinafter referred to as the slurry.

- Except for the perfluoropolyether fluid and the slurry, combine all the remaining ingredients in a vessel equipped with heating and mixing. Heat the remaining ingredients to about 95°C, holding there for a sufficient times until said ingredients are in a liquid form. While maintaining this temperature, stir said ingredients for about 30 minutes, or until obtaining a transparent yellow fluid.  
10 Cool the combination to about 21°C until forming a pale yellow waxy solid; hereinafter referred to as the base.

- Combine the pigment slurry and the base, hereinafter collectively referred to as the first material, with the perfluoropolyether fluid, the second material. Heat the composition to about 90°C until it becomes liquid. Cease heating the composition and mix for about 3 minutes at 10,000 RPM  
15 using a Janke & Kunkel Ultra-Turrax turbine mixer. Pour the composition immediately into a room temperature aluminum bullet mold. Cool the filled mold until the composition is solid. Remove the solid composition from the molds and place in suitable cosmetic packaging.

Example 11:

Ingredient	Weight Percent (%)
Candelilla Wax	1.12
Carnauba Wax	4.27
Ozokerite	6.71
Paraffin	0.94
Acetylated Lanolin	2.37
Isopropyl Isostearate	5.62
Cetyl Alcohol	0.75
Cetyl Lactate	0.75
Octyl Hydroxystearate	3.19

Ascorbyl Palmitate	0.19
Propyl Paraben	0.04
Vitamin E Acetate	0.02
Castor Oil	16.63
Pigments	6.60
Stainers	0.26
Mica	3.34
Isododecane*	12.00
50cSt Dimethicone**	35.20

\*available as Permethyl 99A from Presperse, Inc.

\*\* available as Dow Corning 200 Fluid

Example 11 is made according to the following instructions:

Combine the pigments and castor oil in a vessel with stirring until incorporating the pigments  
 5 into said castor oil. Pass the mixture through a 3-roll mill three times yielding a dispersion of pigment particles in oil; hereinafter referred to as the slurry.

Except for the second material, and the isododecane, combine all the remaining ingredients in  
 a vessel equipped with heating and mixing. Heat the combination to about 95°C, holding there for a  
 sufficient times wherein said ingredients are in a liquid form. While maintaining the heat, stir the  
 10 combination of ingredients for about 30 minutes, obtaining a transparent yellow fluid. Cool the combination to about 21°C until forming a pale yellow waxy solid; hereinafter referred to as the base.

Combine the pigment slurry and the base, hereinafter collectively referred to as the first  
 material, with the second material in order of the highest weight percentage of the composition first.  
 Heat this combination to about 90°C until the combination becomes liquid. Cease heating the  
 15 combination and mix for about 3 minutes at 10,000 RPM using a Janke & Kunkel Ultra-Turrax  
 turbine mixer. Cool the combination to

about 70°C, add the isododecane, and stir for 1 minute using a propeller-type mixer. Pour the combination immediately into chilled (5°C) aluminum bullet molds. Cool the filled molds until the combination is solid. Remove the solid composition from the molds and place in suitable cosmetic  
 20 packaging.

**WHAT IS CLAIMED IS:****1. A lip composition comprising :**

- a. a first materials; and
- b. a second material;

wherein said second material is entrapped within said first material where upon application of said lip composition to the lips said second material separates from said first material forming a barrier layer over said first material deposited on the lips.

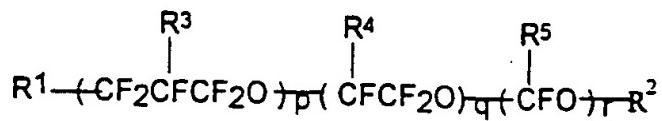
**2. A lipstick composition comprising:**

- a. from 30% to 95%, preferably from 50% to 85%, most preferably from 50% to 60% of a first material whererin said first material is lipophilic and selected from the group consisting of waxes, preferably waxes selected from the group consisting of candelilla, beeswax, beeswax having free fatty acids removed, carnauba, spermaceti, montan, ozokerite, ceresin, paraffin, bayberry, castor waxes, synthetic waxes, microcrystalline waxes, silicone waxes and mixtures thereof; volatile oils, preferably volatile oils selected from the group consisting of hydrocarbon oils, dimethylsiloxanes, cyclic polydimethyl siloxane and mixtures thereof, most preferably volatile oils selected from the group consisting of saturated branched hydrocarbons, unsaturated branched hydrocarbons, saturated linear hydrocarbons unsaturated linear hydrocarbons and mixtures thereof having from 8 to 30, preferably 8 to 14 carbon atoms per molecule; nonvolatile oils, preferably nonvolatile oils selected from the group consisting of caprylic triglycerides, capric triglycerides, isostearic triglyceride, castor oil, adipic triglyceride, dimethicone, octyl dodecanol, oleyl alcohol, hydrogenated vegetable oils, maleated soybean oil, lanolin oil, polybutene, oleyl alcohol; hexadecyl alcohol wheat germ glycerides and mixtures thereof, wherein the oils are compatible with said wax when said wax is liquified; and
- b. from 5% to 70%, preferably from 15% to 50%, most preferably from 30% to 40% of said second material selected from the group consisting of volatile oils having a vapor pressure of less than 10mm Hg, preferably less than 0.5 mm at 30°C, non-volatile materials and mixtures thereof wherein said second material as a viscosity from 0.5 to 5,000 centistokes;

wherein said second material is entrapped in said first material where upon application of said lipstick composition to the lips, said second material separates from said first material forming a barrier layer over said first material deposited on the lips.

3. The composition of Claim 1-2 wherein said second material is optically transparent.
  4. The composition of Claim 1-3 wherein said second material has a surface tension of less than 35 dynes/cm, preferably less than 25 dynes/cm.
  5. The composition of Claim 1-4 wherein said second material comprises a silicone fluid corresponding to the formula:
- $$\begin{array}{c} R_2 \quad R_4 \\ | \quad | \\ R_1 - \text{Si} - O \text{ } y \text{ } Si - R_6 \\ | \quad | \\ R_3 \quad R_5 \end{array}$$
- wherein the end groups  $R_1$  and  $R_6$  are independently selected from the group consisting of hydroxyl groups, lower alkyl groups having carbon chain lengths from  $C_1$  to  $C_6$  and mixtures thereof and the non-end groups  $R_2$ ,  $R_3$ ,  $R_4$  and  $R_5$  are independently selected from methyl groups, fluoroalkyl groups, phenyl groups and mixtures thereof.
6. The composition according to Claim 5 wherein the end groups  $R_1$  and  $R_6$  are methyl groups and  $R_2$ ,  $R_3$ ,  $R_4$  and  $R_5$  are methyl groups.
  7. The composition according to Claim 5-6 wherin said silicone fluid is non-volatile with a viscosity from 1 to 500 centistokes selected from the group consisting of poly(dimethylsiloxane) fluids, poly(phenylmethylsiloxane) fluids, poly(fluoroalkylmethylsiloxane) fluids, the copolymers of said fluids and mixtures thereof, preferably a poly(dimethylsiloxane) fluid, its copolymers and mixtures thereof.
  8. The composition according to Claim 7 wherein said silicone fluid is dimethicone.

9. The composition according to Claim 1-4 whererin the said second material is a perfluoropolyether of general formula:



wherein  $R^1$  though  $R^5$  are selected from the group consisting of fluorine atoms, perfluoroalkyl groups, oxyperfluoroalkyl groups and mixtures thereof, the value of  $p$ ,  $q$ , and  $r$  collectively are of a value such that the perfluoropolyether molecular weight is from 500 to 10,000 wherein  $p$ ,  $q$  and  $r$  may be equal.

10. The composition according to Claim 9 wherein said perfluoropolyether is non-volatile.

## INTERNATIONAL SEARCH REPORT

Int'l Application No  
PCT/US 96/07122

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 A61K7/027 A61K7/025 A61K7/48

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP,A,0 602 905 (REVLON CONSUMER PRODUCTS CORP) 22 June 1994 cited in the application see the whole document ---	1-8
X	US,A,5 288 482 (KRZYSIK DUANE G) 22 February 1994 see example 5 ---	1,5-7
X	FR,A,1 301 966 (LABORATOIRE PHARMACOTECHNIQUE DUBOIS & VINCENT) 16 July 1962 see the whole document ---	1,7
X	EP,A,0 205 961 (ESTEE LAUDER INC) 30 December 1986 see the whole document ---	1,5-8
		-/-

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

\* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

\*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

\*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

\*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

\*&\* document member of the same patent family

Date of the actual completion of the international search

Date of mailing of the international search report

30 September 1996

17.10.96

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax (+31-70) 340-3016

Authorized officer

Sierra Gonzalez, M

## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 96/07122

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP,A,0 494 412 (AUSIMONT SPA) 15 July 1992 see page 1-5, line 52 ---	1-4,9,10
A	PATENT ABSTRACTS OF JAPAN vol. 017, no. 674 (C-1140), 10 December 1993 & JP,A,05 221829 (KOSE CORP), 31 August 1993, cited in the application see abstract -----	1-10

**INTERNATIONAL SEARCH REPORT**

Information on patent family members

 International Application No  
 PCT/US 96/07122

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP-A-0602905	22-06-94	AU-B-	662447	31-08-95
		AU-A-	4870393	30-06-94
		BR-A-	9304975	26-07-94
		CA-A-	2107253	16-06-94
		DE-T-	602905	28-09-95
		ES-T-	2070804	16-06-95
		JP-A-	6199630	19-07-94
		US-A-	5505937	09-04-96
		ZA-A-	9308092	07-06-94
-----				
US-A-5288482	22-02-94	EP-A-	0614658	14-09-94
		JP-A-	6298623	25-10-94
-----				
FR-A-1301966	26-12-62	NONE		
-----				
EP-A-0205961	30-12-86	US-A-	4699780	13-10-87
		AU-B-	577018	08-09-88
		AU-A-	5768386	04-12-86
		CA-A-	1254147	16-05-89
		JP-B-	7045379	17-05-95
		JP-A-	61277606	08-12-86
-----				
EP-A-0494412	15-07-92	CA-A-	2062924	13-09-93
		IT-B-	1244032	28-06-94
		JP-A-	6179809	28-06-94
		US-A-	5368847	29-11-94
-----				